Infrared Spectroscopy Studies of Cyclic Anhydrides as Intermediates for Ester Crosslinking of Cotton Cellulose by Polycarboxylic Acids. IV. *In Situ* Free Radical Copolymerization of Maleic Acid and Itaconic Acid on Cotton

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ABSTRACT: Polycarboxylic acids have been used as crosslinking agents for cotton fabrics and paper to replace the traditional formaldehyde-based reagents. Previously, we found that a polycarboxylic acid esterifies cotton cellulose through the formation of a five-membered cyclic anhydride intermediate. Both maleic acid (MA) and itaconic acid (ITA) are extremely difficult to polymerize under conditions normally used for free radical polymerization. It has been reported in the literature that treatment of cotton fabric with a mixture of MA and ITA significantly improved wrinkle-resistance of the fabric. In this research, we investigated the *in situ* copolymerization of MA and ITA on cotton fabric. Fourier transform-infrared spectroscopy was used to study the anhydride carbonyl formed on the cotton fabric treated with the mixtures of MA and ITA. A redox titration technique also was applied to determine the quantity of alkene double bonds on the treated fabric. It was found that free radical copolymerization of MA and ITA does not occur on the fabric at elevated temperatures when potassium persulfate is present as an initiator. It does occur, however, when both potassium persulfate and sodium hypophosphite are present on the fabric. The *in situ* copolymerization on the cotton fabric probably is initiated by a reduction–oxidation system. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 75: 327–336, 2000

Key words: cellulose; cotton; crosslinking; esterification; free radical polymerization; infrared spectroscopy; *in situ* polymerization; itaconic acid; maleic acid; polycarboxylic acids; redox initiation

INTRODUCTION

In 1988, Welch[1] reported that 1,2,3,4-butanetetracarboxylic acid was able to provide effective crosslinking for cotton cellulose, thus imparting high levels of wrinkle resistance to cotton fabric. Polycarboxylic acids with sodium hypophosphite as a catalyst have been the most promising non-formaldehyde durable press finishes for cotton fabrics.[2–6] Polycarboxylic acids also have been used as crosslinking agents for wood pulp cellulose to improve paper wet strength.[7–10]

In our previous research, we studied the esterification mechanism of cellulose by a polycarboxylic acid and identified the cyclic anhydride intermediates formed by various polycarboxylic acids on the cotton fabric under curing conditions.[11–16]
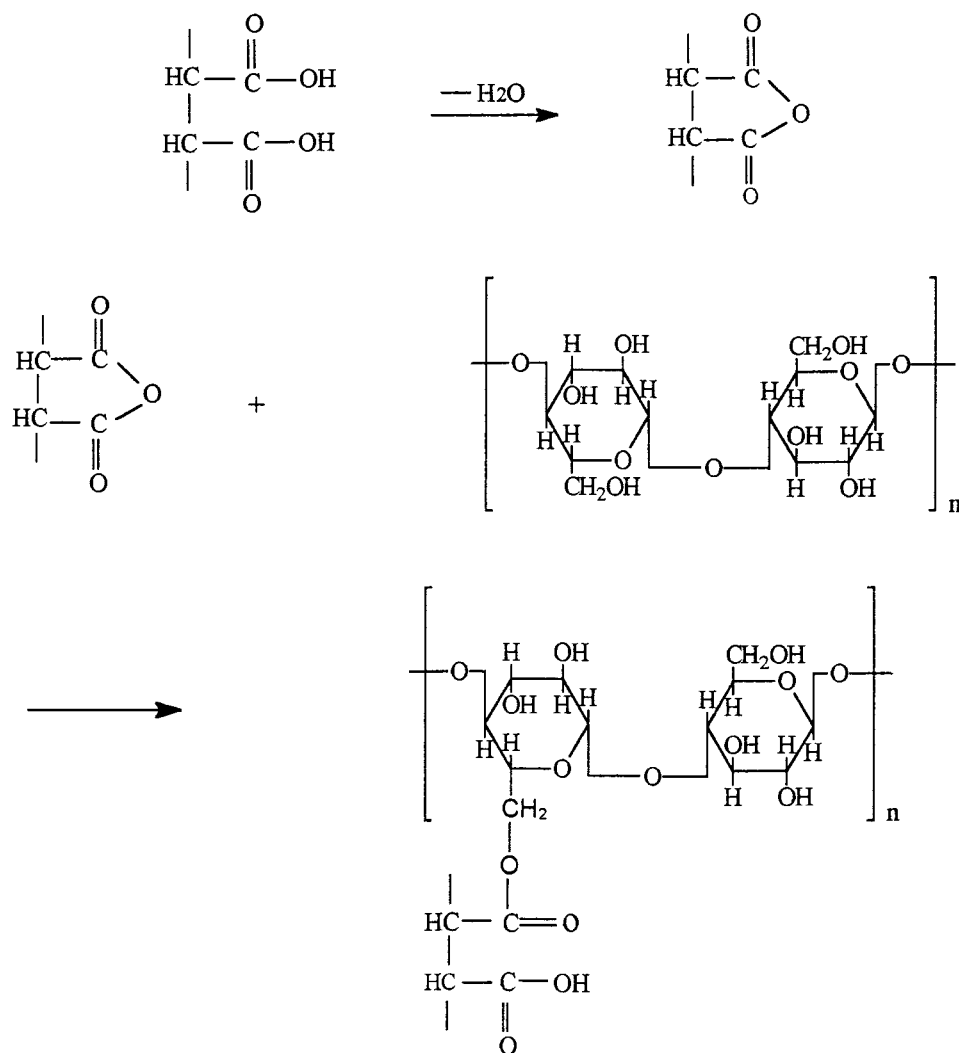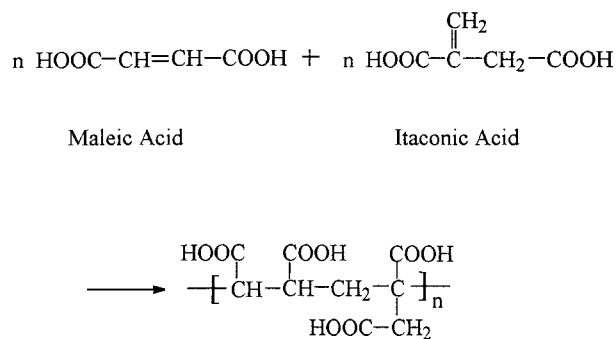
Scheme 1

The esterification of cellulose by a polycarboxylic acid proceeds in two steps: the formation of a cyclic anhydride intermediate by dehydration of two carboxylic acid groups, and the reaction between cellulose and the anhydride intermediate to form an ester (Scheme 1).

Maleic acid (MA) and itaconic acid (ITA) do not polymerize under conditions commonly used for vinyl monomers.[17,18] Copolymers of MA are synthesized by copolymerizing maleic anhydride followed by hydrolysis of the copolymers.[19] In 1992, Choi[20] reported that the use of a mixture of MA and ITA to treat cotton fabric in the presence of potassium persulfate (1.5% of the acids) resulted in substantial increase in the wrinkle resistance of the treated fabric. Bifunctional carboxylic acids are not effective crosslinking agents for cotton cellulose, because they are not able to form the a

five-membered cyclic anhydride intermediate once their first carboxylic acid group esterifies.[13] Choi concluded that MA and ITA copolymerize on the cotton fabric (Scheme 2) and the copolymer of

Scheme 2

MA and ITA become the crosslinking agent for cotton.[20] However, no experimental evidence to support this hypothesis was reported.

The carbonyl of the anhydrides formed by saturated and unsaturated polycarboxylic acids absorb at different frequencies.[13] In this research, we used Fourier transform-infrared (FTIR) spectroscopy to study the anhydride carbonyls on the fabric formed under different conditions so as to elucidate the *in situ* polymerization of MA and ITA.

EXPERIMENTAL

Materials

The cotton fabric used was a desized and bleached print cloth (Testfabrics Style 400). Poly(maleic acid) (PMA) was an aqueous solution with 50% solid content supplied by FMC Corporation. MA, ITA, sodium hypophosphite, monosodium phosphate, and potassium persulfate were supplied by Aldrich.

Fabric Treatment

The cotton fabric was first impregnated in a finish solution containing the polycarboxylic acids, the initiator, and the catalyst. The percent concentration of MA, ITA, and PMA were based on weight (w/w). The impregnated fabric was dried at 85°C for 5 min and then cured at a specified temperature for 2 min. Immediately after the curing process, the cured cotton fabric was analyzed by FTIR spectroscopy for anhydride formation.

Infrared Spectroscopic Measurements

All the infrared spectra were diffuse reflectance spectra collected with a Nicolet 510 FTIR spectrometer and a Specac diffuse reflectance accessory, and are presented as $-\log R/R_0$. Resolution for all the infrared spectra was 4 cm^{-1}, and there were 100 scans for each spectrum. Potassium bromide powder was used as a reference material to produce a background diffuse reflectance spectrum. No smoothing functions and baseline correction were used.

Chemical Analysis

Wet analysis was used to determine the alkene double bond concentration in the cotton fabric treated with MA and ITA. A treated fabric sample was first ground in a Wiley mill to form a powder before analysis. A NaOH solution was added to the powder to convert the unsaturated carboxylic acids to their sodium salts, followed by the addition of a methanol solution saturated with solid NaBr. Quantitative addition reaction of the unsaturated carboxylic acids and Br_2 took place when a standard aqueous Br_2/NaBr solution was added to the mixture. The quantity of the excess amount of Br_2 was then determined by iodometric titration. The percentage (w/w) concentration of the alkene double bond in the treated fabric samples was calculated based on the quantity of Br_2 consumed and the equation described as follows:

Alkene double bond concentration (w/w, %)

$$= \frac{\text{No. of mmoles of } Br_2 \times 24 \times 100}{W}$$

where W is the weight of a sample in milligrams.

The percentage of the remaining free monomers (MA and ITA) on fabric is calculated by the following: [(alkene double bond concentration on the fabric after curing) ÷ (alkene double bond concentration on the fabric before curing)] × 100%.

RESULTS AND DISCUSSION

The infrared spectra of the cotton fabric treated with 6.0% MA is presented in Figure 1(A), in which the band at 1723 cm^{-1} is due to the stretching mode of carboxylic acid carbonyl. Two bands at 1849 and 1778 cm^{-1} due to maleic anhydride emerge in the spectrum when the treated fabric is cured at 140°C for 2 min [Fig. 1(B)]. These two anhydride carbonyl bands are sharp and distinct in the difference spectrum [Fig. 1(C)].

The two carbonyl bands corresponds to the symmetric (in-phase) stretching mode at a higher frequency and the asymmetric (out-of-phase) stretching mode at a lower frequency of a five-membered cyclic anhydride[21,22] (Scheme 3).

The higher frequency band due to symmetric stretching is significantly weaker than the lower frequency one due to asymmetric stretching, because the two carbonyls are in the opposite side of the cyclic system, and the change in the vector sum of the dipole moment of the cyclic —CO—O—CO— system for the symmetric stretching mode is smaller than that for the asymmetric mode. α,β-conjugation reduces the
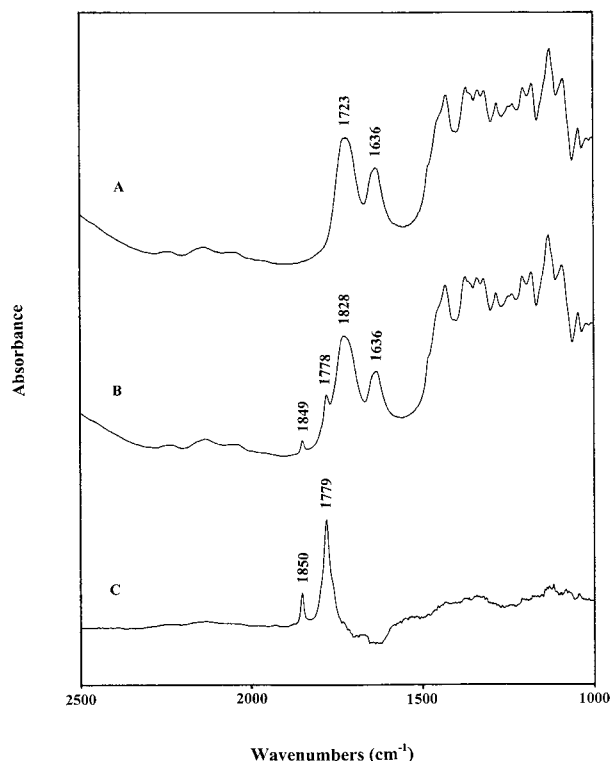
Figure 1 Infrared spectra of (A) the cotton fabric treated with 6.0% MA and dried at 80°C, (B) the cotton fabric thus treated and cured at 140°C for 2 min, and (C) difference spectrum, B − A.

frequency, whereas ring strain increases the frequency.[21] As the ring size increases from five- to six-membered, the cyclic system becomes less symmetric, and the ring strain decreases. Consequently, the two carbonyl bands shifted to lower frequencies and the relative intensity of the symmetric stretching band increases. Frequency and relative intensity of the two carbonyl bands provide a clear indication of the structure of an anhydride.[21,22]

Shown in Figure 2(A, B) are the spectra of the fabric treated with 6.7% ITA before and after

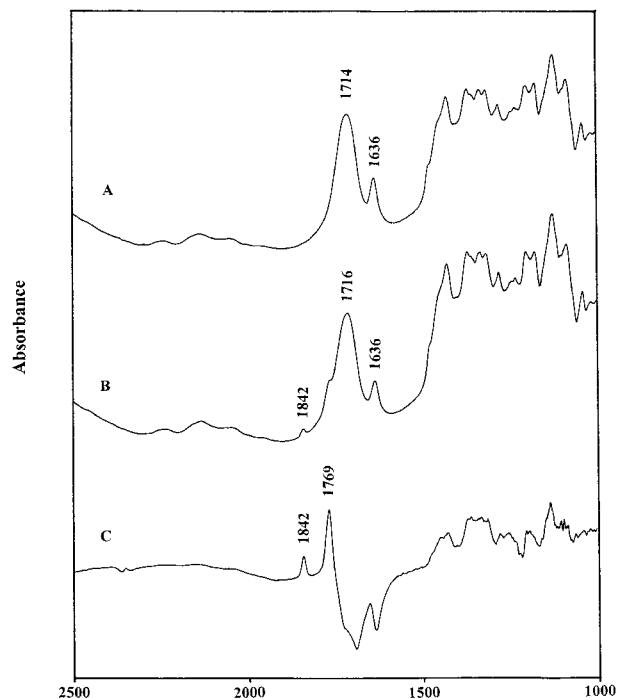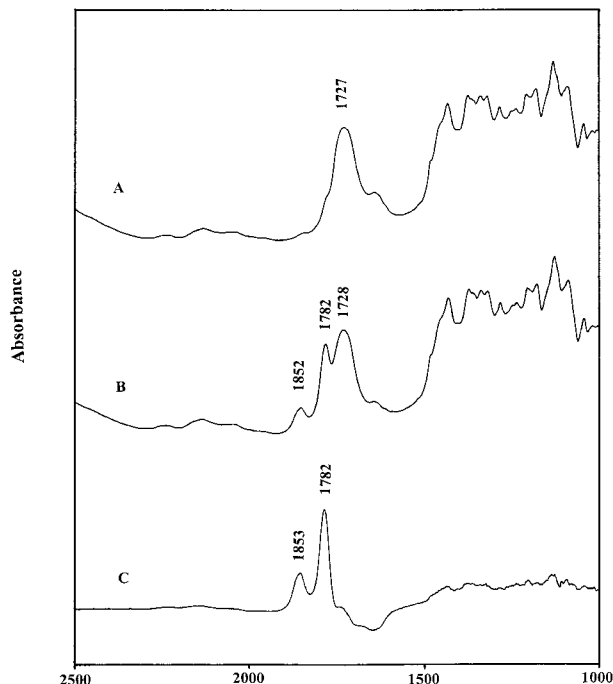Symmetric Stretching   Asymmetric Stretching
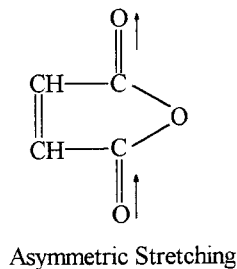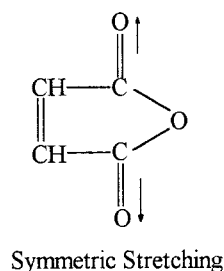
Scheme 3

Figure 2 Infrared spectra of (A) the cotton fabric treated with 6.7% ITA and dried at 80°C, (B) the cotton fabric thus treated and cured at 200°C for 2 min, and (C) difference spectrum, B − A.

curing at 200°C for 2 min, respectively. The two bands at 1842 and 1769 cm^{-1}, due to the anhydride carbonyl formed by ITA, are overlapped by the carboxylic acid carbonyl band at 1716 cm^{-1} in Figure 2(B), but become more distinct in the difference spectrum (Figure 2C). One observes that ITA forms its anhydride at a much higher temperature than MA.

Two explicit carbonyl bands at 1852 and 1782 cm^{-1} due to the five-membered cyclic anhydride are seen in the spectrum of the cotton fabric treated with 6% PMA and cured at 160°C for 2 min [Fig. 3(B)]. These two anhydride carbonyl bands in the difference spectrum [Fig. 3(C)] are more broad than those of MA and ITA shown in Figures 1(C) and 2(C), respectively. In a PMA molecule, an anhydride may form next to another anhydride in the molecular backbone or it may form next to a free carboxyl group. The different chemical environments of the five-membered anhydride intermediates of PMA causes band broadening in their infrared spectra.

The frequencies of the anhydride carbonyl bands of MA, ITA, and PMA formed on the cotton fabric at different temperatures are summarized in Table I. The anhydride of PMA, a saturated
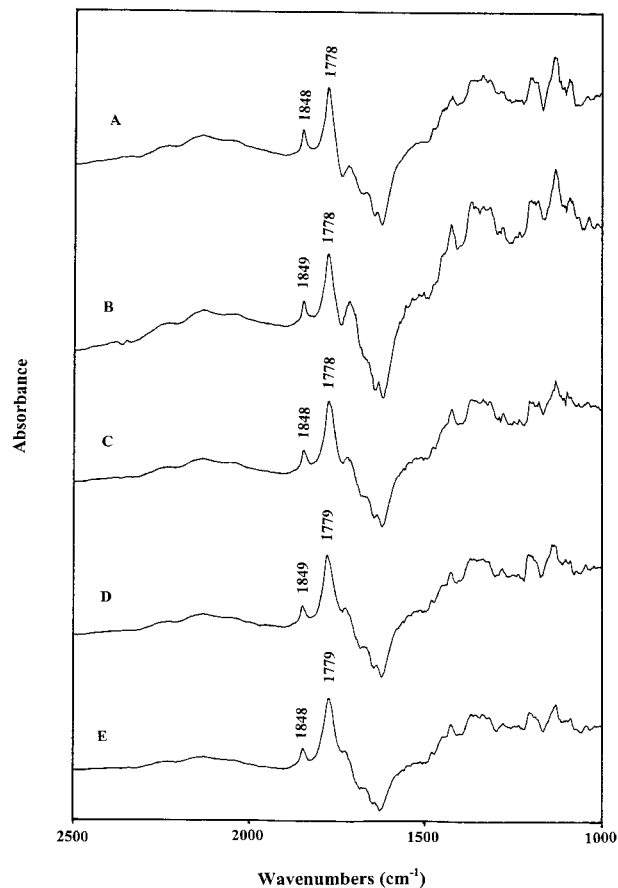
Figure 3 Infrared spectra of (A) the cotton fabric treated with 6.0% PMA and dried at 80°C, (B) the cotton fabric thus treated and cured at 160°C for 2 min, and (C) difference spectrum, B − A.

Figure 4 Difference infrared spectra of the cotton fabric treated with a mixture of 6.0% MA/6.7% ITA in combination with $K_2S_2O_8$ of different concentrations and cured for at 180°C 2 min. The $K_2S_2O_8$ concentration (%): 0.00, 0.25, 0.50, 1.0, and 2.0 (A–E).

polycarboxylic acid, absorbs at a higher frequency than both MA and ITA. The anhydride of MA has higher frequencies for both symmetric and asymmetric modes than the anhydride of ITA. The alkene double bond of MA results in three resonance structures of maleic anhydride. The resonance within the five-membered cyclic system causes it to be coplanar, thus creating higher ring strain than the anhydride of ITA and consequently shifting the two carbonyl bands to higher frequencies.

Table I Frequency of the Anhydride Carbonyl Bands of the Cotton Fabric Treated with 6.0% MA, 6.7% ITA, and 6.0% PMA

Curing Temperature (°C)	MA	ITA	PMA
220	1850/1779	1842/1769	1853/1782
200	1850/1779	1842/1769	1853/1782
180	1850/1779	—	1853/1782
160	1850/1779	—	1853/1782
140	1850/1779	—	1853/1782

The cotton fabric was treated with the mixture of 6% MA and 6.7% ITA in the presence of $K_2S_2O_8$ of different concentrations. All the treated fabric samples were cured at 180°C for 2 min. The difference spectra of these cured fabric samples (the spectrum of the fabric after curing minus that before curing) are presented in Figure 4. The carbonyls of the anhydride formed on the treated fabric absorb at 1849–1848 and 1779–1778 cm^{-1}, and their bands are relatively sharp (Fig. 4). The anhydride seen in the difference spectra appears to be that of MA. When the curing temperature is increased to 200°C and above, the two anhydride bands shift to 1843–1842 and 1771–1769 cm^{-1}, indicating the formation of the anhydride of ITA.

Summarized in Table II are the frequencies of the anhydride carbonyl bands of the cotton fabric treated with 6.0% MA and 6.7% ITA in combination with $K_2S_2O_8$ of different concentrations and

Table II Frequency of the Anhydride Carbonyl Bands of the Cotton Fabric Treated with 6.0% MA and 6.7% ITA in Combination with $K_2S_2O_8$ of Different Concentrations and Cured at Different Temperatures for 2 min

Curing Temperature (°C)	$K_2S_2O_8$ Concentration (%)				
	0	0.25	0.50	1.00	2.00
220	1842/1769	1842/1769	1842/1770	1842/1770	1842/1771
200	1843/1770	1843/1770	1843/1770	1842/1771	1843/1771
180	1848/1778	1849/1778	1848/1778	1849/1779	1848/1779
160	1850/1780	1850/1779	1850/1779	1850/1779	1850/1779
140	1850/1780	1850/1780	1850/1780	1850/1779	1850/1779

cured at different temperatures for 2 min. One observes that the frequency and shape of the anhydride carbonyl band are not affected by the change in $K_2S_2O_8$ concentration (Table II). No anhydrides of saturated polycarboxylic acids are observed on the fabric. Therefore, we can conclude that copolymerization of MA and ITA did not occur on the fabric in the entire temperature range in the presence of $K_2S_2O_8$.

The cotton fabric was treated using the solutions containing 6.0% MA, 6.7% ITA, and 4.0% NaH_2PO_2 in combination with 0.0, 0.25, 0.50, 1.00, and 2.00% of $K_2S_2O_8$. The treated fabric samples were cured at 180°C for 2 min. In the difference spectra (the spectrum of the fabric after curing minus that before curing) demonstrated in Figure 5, one observes that both the frequencies and the shape for the anhydride carbonyl bands changes as the $K_2S_2O_8$ concentration increases from 0.0 to 2.0. The two anhydride carbonyl bands at 1843 and 1771 cm^{-1} in the difference spectra appear to be narrow when no $K_2S_2O_8$ is present in the acids solution or the $K_2S_2O_8$ concentration is 0.25% [Fig. 5(A, B), respectively]. Those two bands are due to the anhydride of ITA. When the $K_2S_2O_8$ concentration is increased to 1.0%, the frequencies of the two anhydride carbonyl bands are shifted from 1843 and 1771 to 1850 and 1779 cm^{-1} [Fig. 5(D)], respectively, and the shape of the 1850 cm^{-1} also is broader than the same band in Figure 4(A, B). Thus, the infrared spectroscopy data indicated that the anhydride formed on the treated fabric is that of a saturated polycarboxylic acid when the $K_2S_2O_8$ concentration is increased to 1.0%.

Shown in Table III are the frequency of the anhydride carbonyl bands of the cotton fabric treated with 6% MA, 6.7% ITA, and NaH_2PO_2 in combination with $K_2S_2O_8$ of different concentra-

tions and cured at different temperatures for 2 min. On the basis of the data presented above, we can conclude that MA and ITA copolymerize only
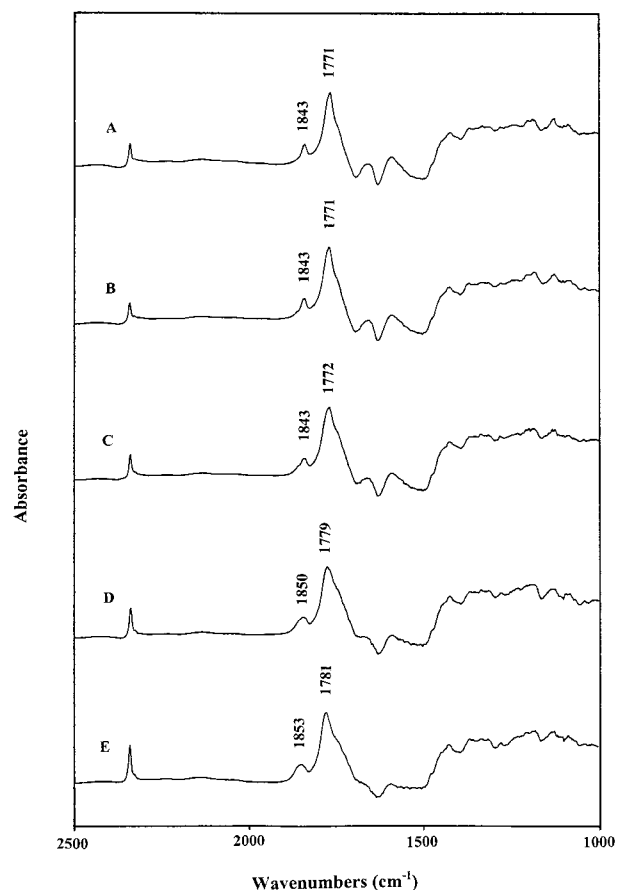
Figure 5 Difference infrared spectra of the cotton fabric treated with a mixture of 6.0% MA, 6.7% ITA, and 4.0% NaH_2PO_2 in combination with $K_2S_2O_8$ of different concentrations and cured at 180°C for 2 min. The $K_2S_2O_8$ concentration (%): 0.00, 0.25, 0.50, 1.0, and 2.0 (A–E).

Table III   Frequency of the Anhydride Carbonyl Bands of the Cotton Fabric Treated with 6.0% MA and 6.7%, 4.0% NaH$_2$PO$_2$ in Combination with K$_2$S$_2$O$_8$ of Different Concentrations and Cured at Different Temperatures for 2 min

Curing Temperature (°C)	K$_2$S$_2$O$_8$ Concentration (%)				
	0	0.25	0.50	1.00	2.00
220	1842/1768	1842/1768	1842/1768	1850/1778	1854/1781
200	1842/1769	1842/1769	1843/1769	1850/1779	1854/1781
180	1843/1771	1843/1771	1843/1772	1850/1779	1853/1781
160	1843/1771	1843/1771	1844/1772	1850/1779	1853/1781
140	1843/1771	1844/1772	1844/1772	1850/1779	1853/1781

when (a) NaH$_2$PO$_2$ is present in the acids solution and (b) the K$_2$S$_2$O$_8$ concentration is relatively high (above 0.5%). One also observes that the *in situ* copolymerization takes place in the temperature range 140–220°C.

It is interesting to note that the amount of K$_2$S$_2$O$_8$ required as an initiator ($>0.5\%$ of the solution or $>4.5\%$ of the monomer) is extremely high compared with that needed in a polymerization process in the liquid state, such as solution or emulsion polymerization. The *in situ* polymerization of MA and ITA takes place in the solid state. When the cotton fabric is impregnated with the MA/ITA solution, the monomers penetrate into the interior of the cotton fibers. Once the fabric is dried, the monomers do not have the same mobility during a polymerization process as those in solution or emulsion polymerization, and consequently a sufficiently large amount of initiator is required for the *in situ* copolymerization to occur.

NaH$_2$PO$_2$ was originally used as a catalyst for the esterification of cellulose by a polycarboxylic acid.[6] Other alkali salts of phosphorous-containing inorganic acids also function as catalysts for the esterification.[6] In order to elucidate the mechanism of the initiation process, we studied the anhydride formation on the fabric treated with 6.0% MA, 6.7% ITA, and 4.0% NaH$_2$PO$_4$ in combination with 0.0, 0.25, 0.50, 1.00, and 2.00% of K$_2$S$_2$O$_8$. The treated fabric was cured at 180°C for 2 min. The difference spectra (the spectrum of the fabric after curing minus that before curing) is presented in Figure 6. One observes that in all cases, the anhydride carbonyl group absorbs at 1842 and 1771 cm^{-1}, and the two bands are sharp. Therefore, it is clear that the anhydride formed on the fabric is the anhydride of ITA. The difference spectra in Figure 6 are in sharp contrast to those in Figure 5, which show changes in

both the band frequency and band shape as the K$_2$S$_2$O$_8$ concentration increases.

Evidently, polymerization of MA and ITA does not take place when NaH$_2$PO$_2$ in the acid solutions is replaced by NaH$_2$PO$_4$. The fact that the copolymerization takes place only when the re-
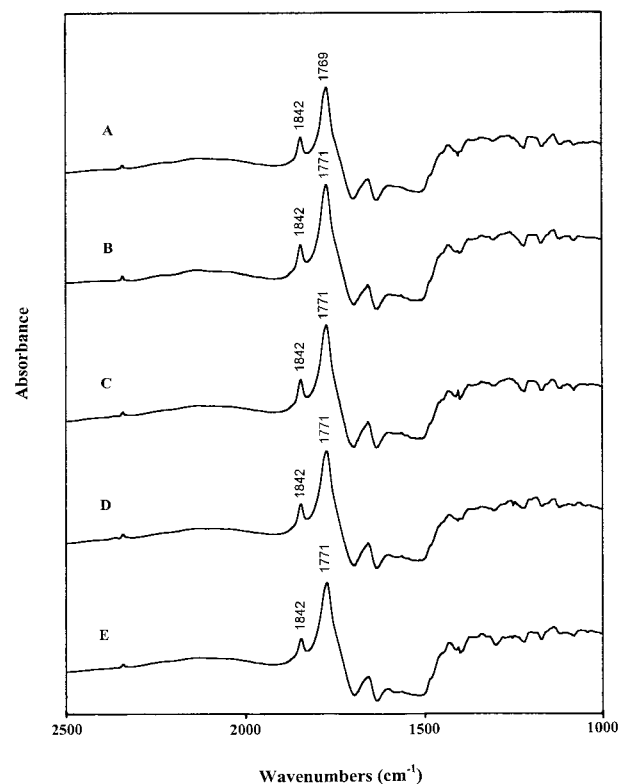
Figure 6 Difference infrared spectra of the cotton fabric treated with a mixture of 6.0% MA, 6.7% ITA, and 4.0% NaH$_2$PO$_4$ in combination with K$_2$S$_2$O$_8$ of different concentrations and cured at 180°C for 2 min. The K$_2$S$_2$O$_8$ concentration (%): 0.00, 0.25, 0.50, 1.0, and 2.0 (A–E).

ducing agent (NaH_2PO_2) is present in the system leads us to believe that the *in situ* copolymerization on cotton is probably initiated by the following redox system:

$$4S_2O_8^{2-} + H_2PO_2^- + 2H_2O \rightleftharpoons$$

$$4SO_4^{\cdot} + 4SO_4^{2-} + PO_4^{3-} + 6H^+$$
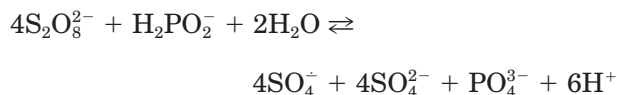
It is possible that when MA or ITA esterifies cellulose, the covalently bonded MA or ITA with a single carboxylic acid group may more readily undergo $S_2O_8^{2-}$-initiated polymerization. We also investigated the formation of the five-membered cyclic anhydride on the treated fabric after hydrolysis. The cotton fabric treated with 6.0% MA, 6.7% ITA, 4.0% NaH_2PO_2, and 2.0% $K_2S_2O_8$ and cured at 180°C for 2 min was washed in hot water ($\sim$ 50°C) with a detergent to facilitate the hydrolysis. After 10 washing cycles, the fabric was finally treated with 0.1 *M* acetic acid to convert the covalently bound carboxylate ions to free carboxylic acid on the fabric. The infrared spectrum of the fabric thus treated is presented in Figure 7(A). The fabric was finally heated at 180°C for 2 min. Two bands at 1850 and 1781 cm^{-1} in the difference spectrum [Fig. 7(C)] are similar to those shown in the difference spectrum of the PMA-treated fabric [Fig. 3(C)]. These two bands in Figure 7(C) are obviously due to the five-membered cyclic anhydride formed by the copolymer of MA and ITA.

When the treated cotton fabric was washed in hot water, the free MA and ITA molecules were removed from the fabric. Those MA and ITA molecules bonded to the cotton fabric through ester linkages were not able to form five-membered cyclic anhydride because each bonded ITA or MA molecule had only one free carboxylic acid group. Only when MA and ITA copolymerized and esterified on the fabric, could they hydrolyze to liberate the free carboxylic acid groups of the copolymer, thus making it possible to form a five-membered cyclic anhydride at elevated temperatures. The formation of the cyclic anhydride on the treated fabric after hydrolysis provides an additional piece of evidence to support the hypothesis that MA and ITA copolymerize on the cotton fabric.

We also removed the copolymer from the fabric using hydrolysis and used a three-angle light-scattering detector to determine the molecular weight of the copolymer. The weight-averaged molecular weight is determined to be (2.014
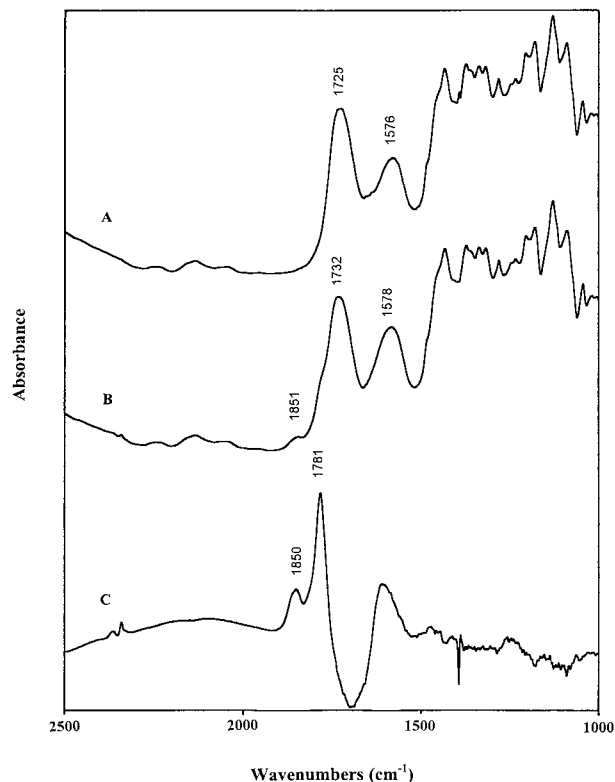

Figure 7 Infrared spectra of (A) the cotton fabric treated with 6.0% MA, 6.7% ITA, 4.0% NaH_2PO_2 and 2% $K_2S_2O_8$, cured at 180°C for 2 min, subject to 10 washing cycles, and finally treated with 0.1 *M* acetic acid, (B) the sample described above, heated at 180°C for 2 min, and (C) the difference spectrum, B − A.

$\pm$ 0.117) $\times 10^3$. The molecular weight study will be discussed in detail in a subsequent paper.

A redox titration technique was used to make a quantitative determination of the concentration of alkene double bond on the treated cotton fabric[23] so as to calculate the amount of free monomer on the fabric after a curing process. The fabric was treated with 6.0% MA, 6.7% ITA, and 4.0% NaH_2PO_2 in combination with $K_2S_2O_8$ of different concentrations and cured at 180°C for 2 min. The percentage of the free monomers on the fabric is presented as function of the $K_2S_2O_8$ concentration of various acid solutions (Fig. 8). The data show that the amount of the free monomers does not change without the presence of $K_2S_2O_8$, and it decreases sharply as the $K_2S_2O_8$ concentration increases (Fig. 8). When the $K_2S_2O_8$ concentration reaches 0.50%, approximately 70% of the monomers copolymerize. Only 18% of MA and ITA remain as monomers when the $K_2S_2O_8$ concentration is 2.0%.
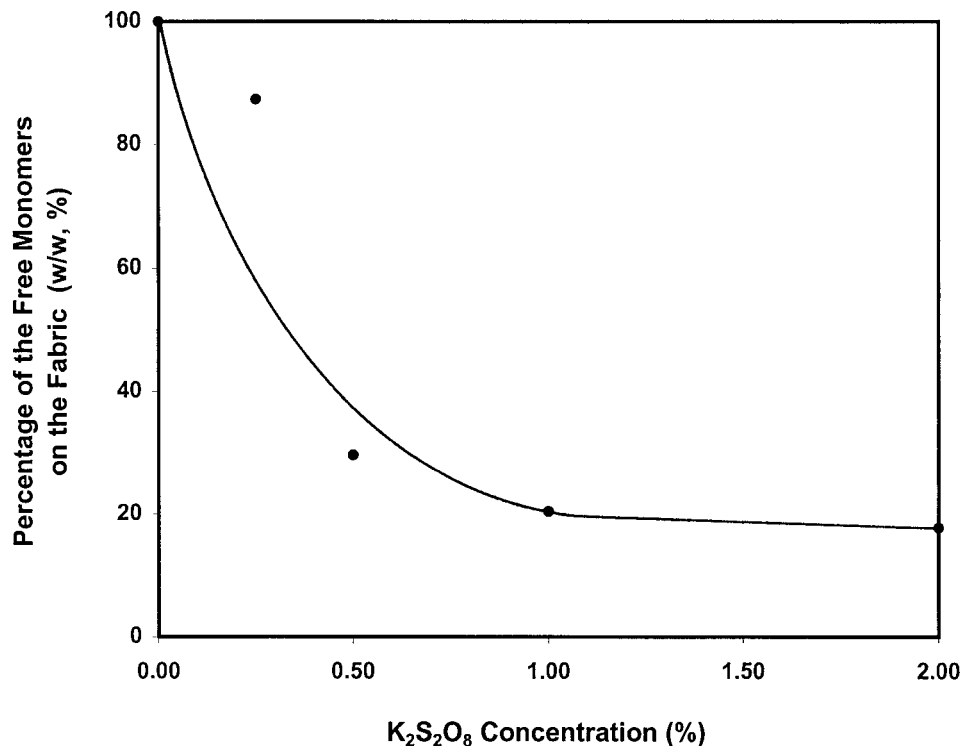
Figure 8 Percentage of the free monomers (w/w) of the cotton fabric treated with 6.0% MA, 6.7% ITA, and 4.0% NaH_2PO_2 in combination with $K_2S_2O_8$ of different concentrations and cured at 180°C for 2 min.

The MA/ITA formulations used to treat cotton fabric reported by Choi[20] contained $K_2S_2O_8$ at 1.5% of the monomers, which was less than 0.2% of the acids solution.[20] Under those conditions, the amount of MA and ITA polymerizing *in situ* on the fabric is small. The data presented in this paper show that a significantly higher concentration of $K_2S_2O_8$ is required for the copolymerization to take place. The performance of the cotton fabric treated with different $K_2S_2O_8$ concentrations also support the observation discussed here and will be reported in another paper.

CONCLUSIONS

Copolymerization of maleic acid and itaconic acid does not occur on the fabric at elevated temperatures when potassium persulfate is present on the fabric as an initiator, but sodium hypophosphite is absent. *In situ* copolymerization does occur, however, when both potassium persulfate and sodium hypophosphite are present on the cotton fabric. The amount of potassium persulfate required for the copolymerization to take place is much higher than that reported in the literature. The *in situ* copolymerization on the cotton fabric is probably initiated by a reduction–oxidation system consisting of potassium persulfate and sodium hypophosphite.

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